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CO_2 CAPTURE AND CONVERSION: A HOMEMADE EXPERIMENTAL APPROACH

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Abstract

During the SARS-2-Covid pandemic our institution sought to continue the teaching and learning of experimental laboratories by designing, assembling, and delivering a microscale chemistry kit to the students' homes. Thanks to this approach students were able to perform ~25 experiments during each one of the Fall 2020 and Spring 2021 semesters in an elective Electrochemistry and Corrosion course offered to Chemical Engineering undergraduates. In addition to performing traditional experiments, students were encouraged to design some of their own and have the entire group reproduce them. One of such student-designed experiments involved the capture of CO_2 and its reduction with a readily available active metal (i.e., Al foil) in aqueous media to generate potentially useful products. The highly negative standard potential of Al is exploited for the reduction of lab-generated CO_2, and the products are chemically tested. Al as a foil has been reported to be electrochemically inactive for carbon dioxide reduction. However, encouraged by an earlier report of the reduction of CO_2 to CO, the Al surface is activated in the present experiment by removal of its natural oxide layer with a solution of $CuCl_2$ produced in an electrochemical cell. This procedure enables Al to react with CO_2 and yield useful chemistry. This experiment turned to be a discovery trip. The detailed procedure is discussed here, as well as the teaching methodology, grading scheme, and student outcomes.

Keywords – Carbon dioxide, Aluminum, Redox reactions, Electrochemistry, CO_2 capture.

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1. Introduction

Distance learning during pandemic- and quasi-normal times has been approached with different strategies including the use of prerecorded videos, virtual and augmented reality, virtual instrumentation, distance-manipulated real instrumentation, simulators, hands-on activities, and the like (Cesin-AbouAtme, Lopez-Almeida, Molina-Labastida & Ibanez, 2021). Each strategy has its own merits and challenges. Our approach consisted of designing a series of experiments to be performed at the students' homes in an Electrochemistry and Corrosion elective course for Chemical Engineering majors in the Fall 2020 and Spring 2021 semesters. This required the assembling of a microscale experimental kit (Ibanez, 2011). To avoid our school's purchasing lag times due to the SARS-Covid pandemic, the instructor purchased all the necessary items in pharmacies, supermarkets, hardware stores, or by internet from his own pocket. Since non-specialized courier companies normally refuse to deliver chemical solutions, he hired a private driver to transport the kits to each student's home. The supplied kit allowed the performance of ~25 distance experiments each semester, many of which were student-designed (e.g., Aguilar-Charfen, Castro-Sayago, Turnbull-Agraz & Ibanez, 2021; Cesin-AbouAtme et al., 2021; Herrera-Loya, Cervantes-Herrera, Gutierrez-Vallejo & Ibanez, 2022). One of such student-designed experiments that was performed by the entire group (Spring 2021), the production and capture of CO_2, is the object of the present paper.

Grades were assigned as follows: 20 %: Student-designed experiments (like the present one), 20 %: Production of a short (3-5 min) video on a selected subject pertaining to the class and its presentation to the group, 40 %: Lab reports (including Title, Objective(s), Materials, Experimental Procedure, Results, Reactions Involved, Conclusions, and Literature References), and 20 %: Final, comprehensive exam.

1.1. Specific Background

The concentration of CO_2 in the atmosphere is a reason for worldwide concern mainly due to its impact on global warming and ocean acidification. Some years ago, it was unlikely that this could surpass the 400-ppm barrier, which is now well exceeded (The Global Carbon Project, 2021). Current mitigation strategies mainly focus on the optimization of energy production from non-carbon intensive sources (e.g., wind and solar power), the development of non-CO_2 producing fuels (e.g., hydrogen), the chemical or physical removal/separation of CO_2 (e.g., chemical reactions, membrane separation, cryogenic distillation), and its utilization for the generation of useful products (e.g., catalytical processes).

Suitable chemical approaches for CO_2 capture and conversion have centered mainly on either a) acid-base, b) redox, or c) coordination chemistries, although futuristic approaches like molecular machine technologies are also being explored (Bushuyev, De Luna, Dinh, Tao, Saur, van de Lagemaat et al., 2018).

2. Design/Methodology/Approach

2.1. Kit Contents

Each kit contained >80 pieces that included: a) basic electronic elements (i.e., resistors, LEDs, a power supply, a multimeter, and alligator clips), b) general lab gear and glassware (i.e., 10- and 50-mL beakers, a 10-mL Erlenmeyer flask, a mini-universal stand with a three-finger clamp, a U-tube, and several auxiliary items as fine-grained sand paper, filter paper, cotton balls, tweezers, toothpicks, rubber bands, and the like), c) electrode materials (i.e., pieces of nichrome, steel, and copper wires, 2-mm ø graphite leads, iron nails, nickel-plated paper clips, Al foil, Mg ribbon, Sn/Pb soldering wire, and Cu-coins), d) volume measurement and transfer gear (i.e., 1-, 10-, and 20-mL needleless syringes, three-way plastic stopcocks, transfer tubing, and a mini funnel), e) solutions and solid reagents (i.e., solid $NaHCO_3$, $MgSO_4$, and $CaCl_2$ packed in small 5-mL glass bottles, as well as solutions of $CuSO_4$, Na_2SO_4, NaOH, HCl, phenolphthalein, and KI contained in 10-mL plastic dropper bottles), and e) safety equipment (i.e., nitrile gloves and safety glasses).

2.2. Teaching Methodology

For each experiment, students were instructed to read the corresponding background in advance and have the necessary components and safety equipment ready in their homes. The first half of each 2-h virtual session was devoted to theoretical issues through a lecture given by the instructor, and the second half to the performance of one experiment per session. The teacher guided each experimental step and all the students performed them simultaneously in front of a web cam so that the teacher could observe their procedures, outcomes, and safety observance. A written report was requested that included the sections described above. The teacher graded the reports and returned them to the students the following week with his comments.

2.3. Experimental

Al as a foil has been reported to be electrochemically inactive for carbon dioxide reduction (Lee, De Riccardis, Kazantsev, Cooper, Buckley, Burroughs et al., 2020). However, encouraged by an earlier report of the reduction of CO_2 to CO (Yoon, Kim, Lee & Sohn, 2018), the Al surface is activated in the present experiment by removal of its natural oxide layer with a solution of $CuCl_2$ produced in an electrochemical cell. $CuCl_2$ is known to etch Al_2O_3, and therefore the next step was to find a suitable home preparation for this chemical. Fortunately, electrochemistry offers a simple way of preparing it from Cu metal and chloride ions as follows.

2.3.1. Surface Etching

A spatula-tip of table salt (NaCl, preferably non-iodized salt to avoid possible interferences from added chemicals) was dissolved in ~ 6 mL of drinking water in a 10-mL beaker. One drop of 4 M HCl was then added. Two 10-cm pieces of thin Cu wire (for example, gauge 22 AWG) were cut and used as electrodes, and were coiled as to offer a high surface area for the electrochemical reaction. Both coils were immersed in the solution, allowing ~ 2 cm for alligator cable connections. Each alligator cable was secured with adhesive tape to a universal support or a similar device to prevent short circuits and the tip-over of the beaker.

Each submerged coil (electrode) was connected to one of the terminals of a 9-V battery. An electrochemical process started immediately. Bubbles were observed at both electrodes, while a blue-greenish coloration started developing in the surroundings of the positive electrode (Anode). After a couple of minutes of the onset of such coloration, both terminals were disconnected. A folded, ~10 cm^2 piece of kitchen Al foil was immersed in the solution for a few minutes. This process removed the Al_2O_3 layer and part of the Al metal, and this activated foil was then ready for reaction with CO_2. The foil was taken out and rinsed with water.

2.3.2. CO_2 Production

The treated foil was submerged in a 50-mL beaker half-filled with drinking water. It was covered with a piece of kitchen transparent film food wrap (e.g., Reynolds Sure Seal). A CO_2 generator was prepared by allowing ~ 0.1 g of kitchen $NaHCO_3$ to react with 4 mL of cooking vinegar in a 20-mL syringe, using Mattson's method (Mattson, 2017).

2.3.3. CO_2 Bubbling and Reaction

Once the CO_2 was generated and transferred to a clean syringe, a hose was connected to the tip of the syringe, passed under the film wrap into the beaker, and the other end of the hose was pointed towards the Al foil. (The use of an automatic delivery pipet tip at the end of the hose yields better results by producing finer bubbles). The plunger was then slowly pushed as to produce a reasonably constant stream of gas impinging the Al surface. A slow generation of a white suspension indicated that the CO_2 was reduced and a solid reduction product generated (A loupe or a laser to prove the Tyndall effect helps). The hose was removed from inside the beaker, which was then closed with transparent film wrap. The suspension was allowed to stand still for half a day with the CO_2 atmosphere above it. This promoted the formation of a more abundant white precipitate. More precipitate was obtained when this procedure was repeated two or three times.

3. Results and Discussion

$CuCl_2$ etches both, the Al_2O_3 protective layer and the Al metal (Annamalai & Hiskey, 1978; Poinern, Ali & Fawcett, 2011). For Cu(II) to exist in solution, either a circumneutral or an acidic pH is needed. We opted for the second case by adding 1 drop of 4 M HCl. By applying a potential with a 9-V battery, the oxidation of Cu and Cl^- is promoted. Chlorine gas is observed to evolve at the anodic surface. This gas also attacks Cu and reinforces the production of the desired $CuCl_2$, as signaled by a bluish-green coloration around the anode and a visible surface attack.

After being partially submerged in the freshly produced $CuCl_2$ solution for a couple of minutes, the Al foil develops an easily distinguishable portion where the Al_2O_3 layer has been removed and therefore the Al surface activated. See Figure 1.

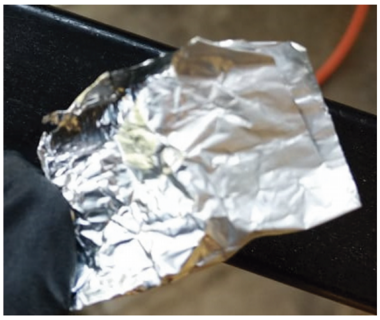

Figure 1. Al foil: etched (left) and non-etched (right)

When the Al foil is completely submerged for etching during a few minutes, the etching process forms $AlCl_3$ from the dissolution of the oxide and the metal. This activated foil then reacts with the bubbled CO_2 whose reduction can follow different paths depending on the working potential and solution conditions (Rajeshwar & Ibanez, 1997; Sánchez-Sánchez, 2004).

Since oxidized surfaces are highly selective towards C_2 products (Varela, 2020), the likely product is oxalic acid:

$$2CO_2 + 2H^+ + 2e^- \rightleftharpoons (COOH)_2 \tag{1}$$

that in the presence of Al(III) ions forms the white insoluble aluminum oxalate. See Figure 2.

$$2Al_{(s)} + 6CO_2 + 6H^+_{(aq)} \rightleftharpoons 3H_2C_2O_{4(s)} + 2Al^{3+}_{(aq)} \qquad E^o = 1.195\ \mathrm{V} \tag{2}$$

(Standard potentials for the calculation are taken from Bard, Parsons and Jordan, 1985).

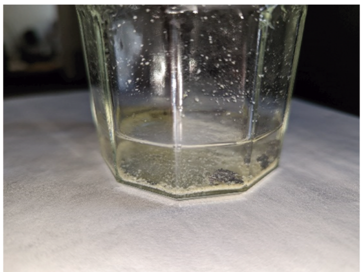

Figure 2. White precipitate obtained from the bubbling of CO_2 on an activated Al surface

If the suspension is then concentrated by evaporating approximately half of the water present through heating over an iron skillet in a kitchen stove (without boiling, for 20-30 min), the presence of the precipitate becomes much more obvious after allowing the solution to cool down to room temperature. If this resulting mixture is allowed to stand still for some 2 days at room temperature, water evaporates completely. The result includes highly corroded aluminum foil pieces and white crystals attributed to insoluble aluminum oxalate. See Figure 3.

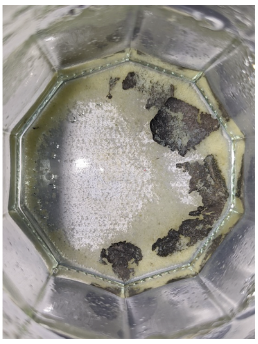

Figure 3. Crystals (white) and corroded Al foil (grey) obtained upon drying of the suspension resulting from the CO_2 reduction with Al

The possible composition of the precipitate can be discussed with the students, since several white precipitates other than aluminum oxalate are possible including NaCl, $AlCl_3$, Al_2O_3, and other salts from the water utilized. The student team that designed the experiment was encharged of the following qualitative analytical procedures to discern the chemical nature of this precipitate, which required further chemicals. To ensure that the precipitate was not a carbonate, HCl was added to it. The absence of bubbles and the fact that the precipitate was not affected by the acid, corroborated the absence of a carbonate. To test for the presence of a reducing group (i.e., oxalate) in the precipitate, a solution of $KMnO_4$ was added to a portion of the precipitate (Karamad, Khosravi-Darani, Hosseini & Tavasoli, 2019). A discoloration effect was observed, which is further evidence of the presence of an oxalate. See Figure 4. This discards non reducing aluminum compounds such as $AlCl_3$, $Al_2(CO_3)_3$, and Al_2O_3.

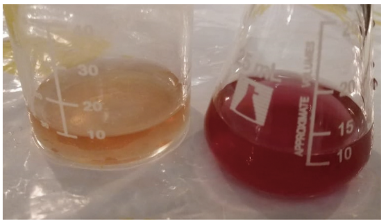

Figure 4. Resulting solution (left) from the reduction of a dil. $KMnO_4$ solution in a circumneutral medium (right) by the white precipitate obtained from the reduction of CO_2

To further test our hypothesis of the chemical nature of the precipitate, i. e., aluminum oxalate, $Al_2(C_2O_4)_3$ a qualitative test using aluminon (ammonium aurintricarboxylate) yielded the expected red complex shown in Figure 5 due to the presence of Al(III) (Bertsch, Alley & Ellmore, 1981; Clark & Krueger, 1985). For this, we placed 10 mL of drinking water in a 25-mL Erlenmeyer flask and then added two drops of 4 M HCl, plus a small aluminon crystal. The mixture was stirred to complete dissolution. We then added 10 mL of drinking water to a 50-mL beaker, poured the contents of the Erlenmeyer flask into the beaker, stirred, and observed any color changes.

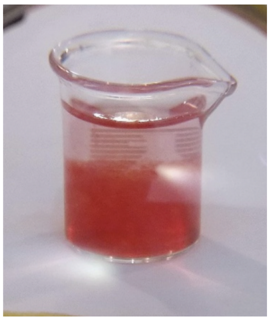

Figure 5. Formation of a red colored Al(III)-aluminon complex

Additional variable control tests were performed to further discard alternative interactions. The experiment was tested with three different variations: a) removing the presence of Al altogether from the experiment, b) skipping the pretreatment of the Al foil, and c) skipping the CO_2 atmosphere addition. In none of these three variations was any precipitate observed, even after a few hours. This confirms that all three compounds and conditions are necessary to obtain the observed results.

We then analyzed key learning outcomes from this course through a voluntary survey applied at the end of the semester (responded anonymously by 92% of the students, i.e., 11 out of 12). The main outcomes are now presented in Table 1 (Ibanez & Contreras-Ruiz, 2021) and they correspond to the following experimental areas: Basic electrical circuits and measurements, redox reactions and Pourbaix diagrams, energy generation and storage, metal deposition (electro- and electroless), environmental electrochemistry, water electrolysis, organic electrochemistry, and organic electrochemistry.

Responses and percentages	**Strongly Agree**	**Agree**	**Undecided**	**Disagree**	**Strongly Disagree**
Specific knowledge acquired during the course					
With the practice at home I improved my mastery of the topics on environmental, organic, inorganic electrochemistry, energy storage, electrochemical theory, and corrosion	45	55	0	0	0
I learned to design sound scientific procedures to generate new experiments or solve basic experimental problems.	82	18	0	0	0
I understood and applied green chemistry principles such as decreasing the amounts of substances used and minimizing the generation of waste.	55	45	0	0	0
Using scientific data published in reliable sources helped me better understand or report the results I observed.	45	36	18	0	0
I learned the correct use of basic glassware and laboratory equipment following the proper techniques.	91	9	0	0	0
I discussed with my classmates and with the professor the scientific or mathematical theories and models developed through data analysis to explain some of the observed phenomena.	18	45	36	0	0
Competencies developed during the course					
I further developed scientific reasoning, particularly in electrochemistry and corrosion.	55	45	0	0	0
I generated oral and written arguments consistent with scientific practice.	55	45	0	0	0
I made progress in my ability to prepare laboratory reports and audiovisual resources aligned with good scientific practices.	73	27	0	0	0
I managed my time and space efficiently in my home laboratory, and I improved in developing teamwork skills.	45	36	18	0	0
I understood and applied the principles of ethical behavior in academia and in scientific practice.	82	9	9	0	0
I developed learning and autonomous study skills applied to electrochemistry and corrosion.	55	45	0	0	0
Safety skills practiced during the course					
I knew and used the safety standards and the proper handling of chemicals in a home laboratory.	100	0	0	0	0
I participated in chemical practice in a conscious manner regarding safety and the environment.	82	18	0	0	0

Table 1. Student outcomes

Lastly, in this survey essentially all the students said that they appreciated the passion and efforts of the teacher and considered this as a source of motivation, together with his ample knowledge of the subject.

4. Conclusions

A homemade experiment can demonstrate that CO_2 is reduced at an active metal surface (i.e., Al foil) to generate useful chemistry. A chemical etchant (i.e., $CuCl_2$) is electrochemically prepared with the aid of a 9-V battery. The white solid generated responds to four physical and chemical properties of aluminum oxalate. The experiment generated a series of positive student learning outcomes.

5. Hazards and Treatment of Residues

Wear eye and hand protection at all times. HCl is corrosive to the skin and eyes. $CuCl_2$ is corrosive, acute toxic, irritant, and an environmental hazard. Aluminon is an irritant. $AlCl_3$ is corrosive and irritating to the eyes, skin, and mucous membranes. The residues from the CO_2 production present no hazard and can be discarded down the drain. The residues from the $KMnO_4$ test, the $CuCl_2$ etching solution, and the aluminon test must be confined as metallic residues. The electrochemical part of this experiment must be conducted in a well-ventilated space since small amounts of toxic Cl_2 gas are generated.

Declaration of Conflicting Interests

The authors declared no potential conflicts of interest with respect to the research, authorship, and/or publication of this article.

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